Validation Worksheets – Semivolatiles

Site:_____ QC Level:_____ Project No.:_____

SDG#_____ No. Samples_____ Matrix_____ Lab:_____

Attach Copy of Case narrative, Lab Sample ID pages, and Flagged Data Tables

Analysis Performed (check all that apply): ☐ SW-846 ☐ CLP ☐ SIM ☐ T ES/ER/TM-16 ☐ TO-14 ☐ Y/P65-9006 ☐ 624

Parameter	Criteria	Acceptable	Not Acceptable
Holding Time; Preservative	**H_2O** - 7 days to extraction and 40 days to analysis **Soil** – 14 days to extraction and 40 days to analysis 4°C (+ 2°C)		
DFTPP GC/MS Instrument Performance Check (Tuning) (does not apply to SIM technique)			
Requirement	Up to 50 ng Decafluorotriphenylphosphine (DFTPP) on-column		
Frequency	Every 12 hours		
Criteria	51 10.0 - 80.0% of mass 198 68 Less than 2% of mass 69 69 Present 70 Less than 2% of mass 69 127 10 – 80% of mass 69 197 Less than 2% of mass 198 198 Base peak, 100% relative abundance 199 5 – 9% of mass 198 275 10 – 60% of mass 198 365 Greater than 1% of mass 198 441 Present, by less than mass 443 442 50 – 100% of mass 198 443 15 – 24% of mass 442		
Initial Calibration:			
Requirement	All target analytes and Deuterated Monitoring Compounds (DMCs)		
Levels	5.0, 10, 20, 40, & 80 ng/uL for all compounds with the following exception 10.0, 20, 40, & 80 ng/ul for 2,4-dinitrophenol; pentachlorophenol; 2nitroaniline; 3-nitroaniline; 4-nitroaniline; 4-nitrophenol, and 4,6-dinitro-2-methylphenol 0.10, 0.20, 0.40, 0.80, & 1.0 ng/uL for the SIM method 0.20, 0.40, 0.80, & 1.0 ng/uL for Pentachlorophenol by the SIM method		

Parameter	Criteria		Acceptable	Not Acceptable
Frequency	Within 12 hours of the associated instrument performance check, each time instrument is set up and after CCV failure			
Criteria	RRF $\geq$ 0.01 for poor performers and associated DMCs and $\geq$ 0.05 for remaining analytes Correlation Coefficient $\geq$ 0.995 %RSD of all RRFs $\leq$ 40% for poor performers and associated DMCs and $\leq$ 20% for remaining analytes			
Criteria (cont.)	**Poor Performers**			
	2,2'-Oxybis-(1-hloropropane) p-chloroaniline Hexachlorobutadiene Hexachlorocyclopentadiene 2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol Acetophenone Caprolactam Atrazine Di-n-butylphthalate	Bis(2-ethylhexyl) phthalate Benzaldehyde 4-Nitroaniline 4,6-Dinitro-2-methylphenol N-Nitrosodiphenylamine 3,3`-Cichlorobenzidine 1,1-Biphenyl Dimethylphthalate Diethylphahtalate 1,2,4,5-Tetrachlorobenzene Carvazole Butylbanzylphthalate Di-n-octylphthalate		
CCV Standards				
Level	All target analytes and DMCs			
Frequency	Beginning and end of each 12-hour analysis period following the analysis of the instrument performance check and prior to analysis of method blank and samples			
Criteria	**Opening CCV** – RRF for poor performers and associated DMCs $\geq$ 0.01, all other remaining analytes $\geq$ 0.05. **Closing CCV** – All RRFs $\geq$ 0.01. **%D** between initial calib. and opening CCV for poor performers and associated DMCs $\leq$ 40%, all other remaining analytes $\leq$ 25%. **%D** between initial calib. and closing CCV must be $\leq$ 50% for all analytes.			
Blanks				
Level	No contaminants should be found in any blank.			
Frequency	**CCB –** After calibration standards and CCVs **Method Blank** – Once for every 20 samples per matrix.			
Criteria	**Low level** – < CRQL (BEHP < 5X CRQL) **Medium level** - < CRQL All **non-target** analytes must be < 10 ug/L			
Deuterated Monitoring Compounds –Surrogates (DMCs)				
Frequency	Spiked in all samples			

Parameter	Criteria					Acceptable	Not Acceptable
Criteria	Please see attached tables						
Field Duplicate Samples							
Level	Cannot use blank or PE sample						
Frequency	1 DUP per matrix per 10 samples (10%)						
Criteria	**Aqueous:** 20% RPD						
	Soil: 35% RPD						
Matrix Spike Samples							
Level	Cannot use blank or PE sample						
Frequency	1 MS/MSD per matrix per 20 or fewer samples (or 5%)						
Criteria	**Compound**	**%R H2O**	**%R Soil**	**RPD H2O**	**RPD Soil**		
	Phenol	12-110	26-90	0-42	0-35		
	2-chlorophenol	27-123	25-102	0-40	0-50		
	N-nitroso-di-n-propylamine	41-116	41-126	0-38	0-38		
	4-Chloro-3-methylphenol	23-97	26-103	0-42	0-33		
	Acenaphthalene	46-118	31-137	0-31	0-19		
	4-Nitrophenol	10-80	11-114	0-50	0-50		
	2,4-Dinitrotoluene	24-96	28-89	0-38	0-47		
	Pentachlorophenol	9-103	17-109	0-50	0-47		
	Pyrene	26-127	35-142	0-31	0-36		
Gel Permeation Chromatography (GPC) Cleanup							
Frequency	GPC is used for the cleanup of all non-aqueous sample extracts and for aqueous sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.						
	At least once every seven (7) days, the calibration of the GPC unit must be checked by injecting the calibration solution.						
Criteria	Peaks must be observed and symmetrical for all compounds in the calibration solution.						
	Corn oil and the phthalate peaks exhibit greater than 85% resolution.						
	The phthalate and methoxychlor peaks exhibit greater than 85% resolution.						
	Methoxychlor and perylene peaks exhibit greater than 85% resolution.						
	Perylene and sulfur peaks must not be saturated and should exhibit greater than 90% baseline resolution.						
	The Retention Time (RT) shift is less than 5% between UV traces for bis(2-ethylhexyl)phthalate and perylene.						
	A GPC blank must be analyzed after each GPC calibration and is acceptable if the blank does not exceed the Contract Required Quantitation Limits (CRQL) for any target analytes, except for bis(2ethylhexyl)phthalate, which must be less than 5x the CRQL.						
Internal Standards							

Parameter	Criteria	Acceptable	Not Acceptable
Frequency	Included in EVERY sample		
Criteria	**Area Counts** of Samples, MS/MSDs, blanks and PEs must not vary more than 50 – 200% from assoc. CCV or midpoint initial calib. standard. **Retention Time** of internal standard must not vary more than ±30 seconds from the RT of the internal standard in the assoc. CCV or midpoint initial calib. standard.		
Target Compound Identification			
Criteria	Relative retention times must be within ± 0.06 RRT units of the standard RRT (opening CCV or midpoint initial calib. standard. All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum. The relative intensities of these ions must agree within ±20% between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%). Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.		
Compound Quantitation and Reported CRQL			
Criteria	Compound quantitation, as well as the adjustment of the CRQLs and percent moisture, must be calculated according to the correct equation.		
Criteria	Compound Relative Response Factors (RRFs) must be calculated based on the internal standard associated with that compound, as listed in the method. Quantitation must be based on the quantitation ion (m/z) specified in the method for both the internal standards and target analytes. The compound quantitation must be based on the average RRF from the associated initial calibration		
Tentatively Identified Compunds			
Criteria	For each sample, the laboratory must conduct a mass spectral search of the NIST/USEPA/NIH (May 2002 release or later), and/or Wiley (1998 release or later), or equivalent mass spectral library, and report the possible identity for 30 of the largest volatile fraction peaks which are not DMCs, internal standards, or target compounds, but which have an area or height greater than 10% of the area or height of the nearest		

Parameter	Criteria	Acceptable	Not Acceptable
	internal standard. Estimated concentrations for TICs are calculated similarly to the Target Compound List (TCL) compounds, using total ion areas for the TIC and the internal standard, and assuming a RRF of 1.0.		

Level IV/D/Definitive Data with Raw Data

Note: calculations/transcriptions may be written down in raw data package or on separate paper as proof that they were performed.

YES	NO	
☐	☐	Transcriptions checked for 10% of the data (raw vs. summaries)?
☐	☐	Quantitation calculations checked for 10% of ALL data (i.e. calibrations, check standards, spikes, dups, results)?

Reviewers Signature:

_____ Date____/____/____

Semivolatile Deuterated Monitoring Compound (DMC) and Recovery Limits

DMC	Recovery Limits (%) for Water Samples	Recovery Limits (%) for Soil/Sediment Samples
Phenol-d5	39 - 106	17 -103
Bis-(2-chloroethyl) ether-d8	40 - 105	12 - 98
2-Chlorophenol-d4	41 - 106	13 - 101
4-Methylphenol-d8	25 - 111	8 - 100
Nitrobenzene-d5	43 - 108	16 - 103
2-Nitrophenol-d4	40 - 108	16 - 104
2,4-Dichlorophenol-d3	37 - 105	23 - 104
4-Chloroaniline-d4	1 - 145	1 - 145
Dimethylphthalate-d6	47 - 114	43 - 111
Acenaphthylene-d8	41 - 107	20 - 97
4-Nitrophenol-d4	33 - 116	16 - 166
Fluorene-d10	42 - 111	40 - 108
4,6-Dinitro-2-methylphenol-d2	22 - 104	1 - 121
Anthracene-d10	44 - 110	22 - 98
Pyrene-d10	52 - 119	51 - 120
Benzo(a)pyrene-d12	32 - 121	43 - 111
Fluoranthene-d10 (SIM)	50 - 150	50 - 150
2-Methylnaphthalene-d10 (SIM)	50 - 150	50 - 150

Semivolatile Deuterated Monitoring Compounds (DMCs) for Selective Ion Monitoring (SIM) and theAssociated Target Compounds

Fluoranthene-d10 (DMC)	2-Methylnapthalene-d10 (DMC)
Fluoranthene	Napthalene
Pyrene	2-Methylnapthalene
Benzo(a)anthracene	Acenapthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	

Semivolatile Deuterated Monitoring Compounds (DMCs) and the Associated Target Compounds

Phenol-d5 (DMC)	**2-Chlorophenol-d4 (DMC)**	**2-Nitrophenol-d4 (DMC)**
Benzaldehyde Phenol	2-Chlorophenol	Isophorone 2-Nitrophenol
bis(2-Chloroethyl) ether-d8 (DMC)	**4-Methylphenol-d8 (DMC)**	**4-Chloroaniline-d4 (DMC)**
bis-(2-Chloroethyl) ether 2,2'-oxybis(1-Chloropropane) bis(2-Chloroethoxy) methane	2-Methylphenol 4-Methylphenol 2,4-Dimethylphenol	4-Chloroaniline Hexachlorocyclopentadiene 3,3'-Dichlorobenzidine
Nitrobenzene-d5 (DMC)	**2,4-Dichlorophenol-d3 (DMC)**	**Dimethylphthalate-d6 (DMC)**
Acetophenone	2,4-Dichlorophenol	Caprolactam
N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine	Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol	1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate bis(2-ethylhexyl)phthalate Di-n-octylphthalate
Fluorene-d10 (DMC)	**Anthracene-d10 (DMC)**	**Pyrene-d10 (DMC)**
Dibenzofuran Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole	Hexachlorobenzene Atrazine Phenanthrene Anthracene	Fluoranthene Pyrene Benzo(a)anthracene Chrysene
Acenaphthylene-d8 (DMC)	**4-Nitrophenol-d4 (DMC)**	**Benzo (a) pyrene-d12 (DMC)**
Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene Acenaphthylene Acenaphthene	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol-d2 (DMC)		
4,6-Dinitro-2-methylphenol		